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US 4906083 A

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(54) Abstract Title
Marking diamond using ion beam, laser or plasma to produce a diffraction effect

(57) The surface of a diamond gemstone is marked with an alphanumeric character in the form of parallel grooves 10 using a focussed ion beam (eg gallium), excimer laser or plasma etching. The mark cannot be read with the naked eye, the grooves producing a visible diffraction effect such that the mark can be read under magnification. The marked diamond 104 may be viewed using a microscope when illuminated by two directional light sources 106 at an angle corresponding to the angle of diffraction.

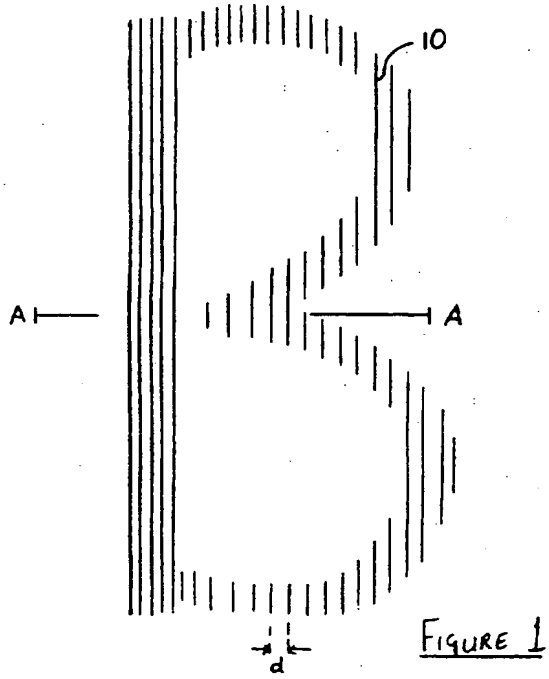

FIGURE 1

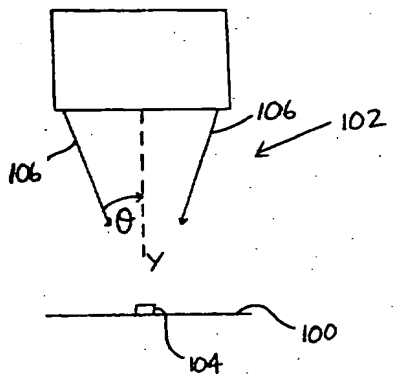

FIGURE 3

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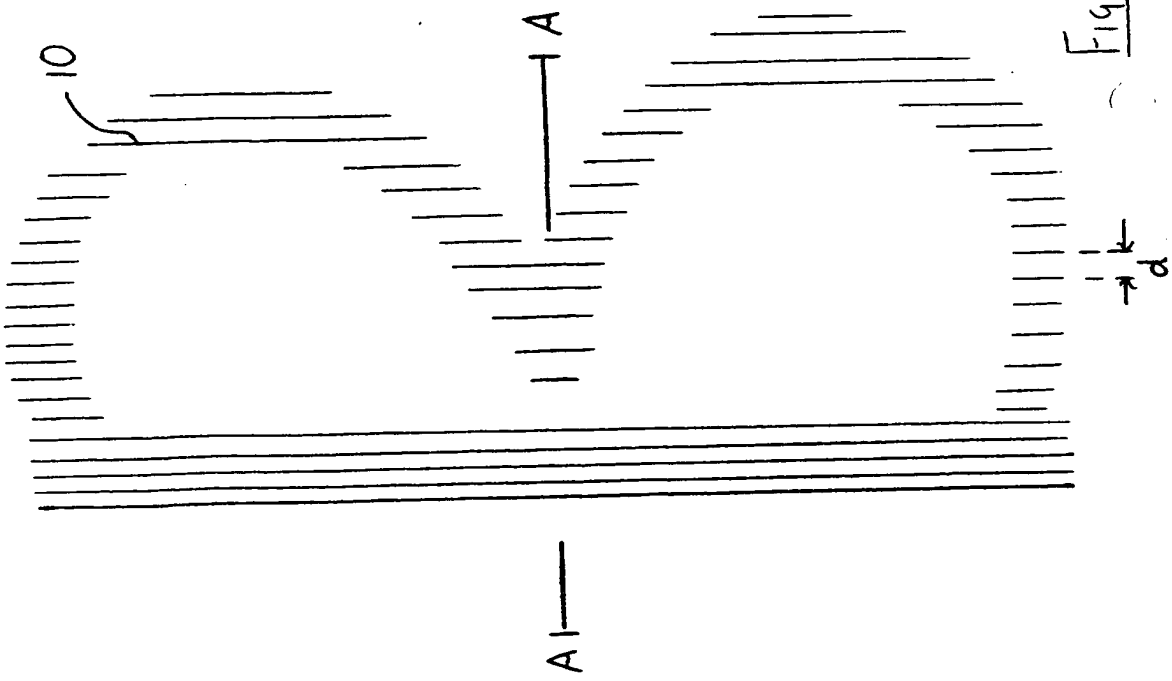

FIGURE 1

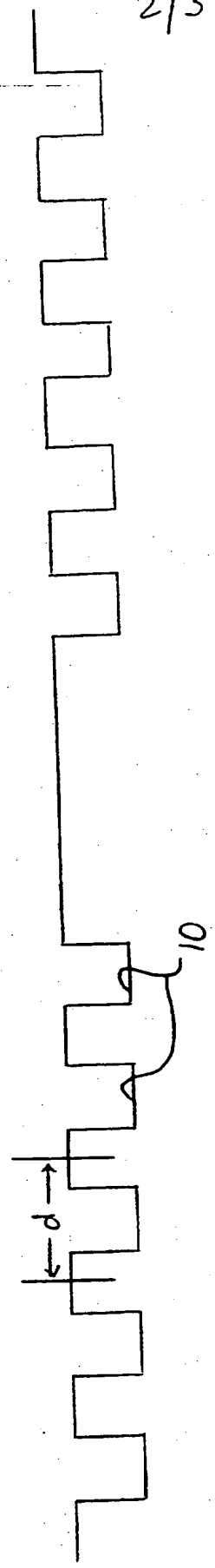

FIGURE 2

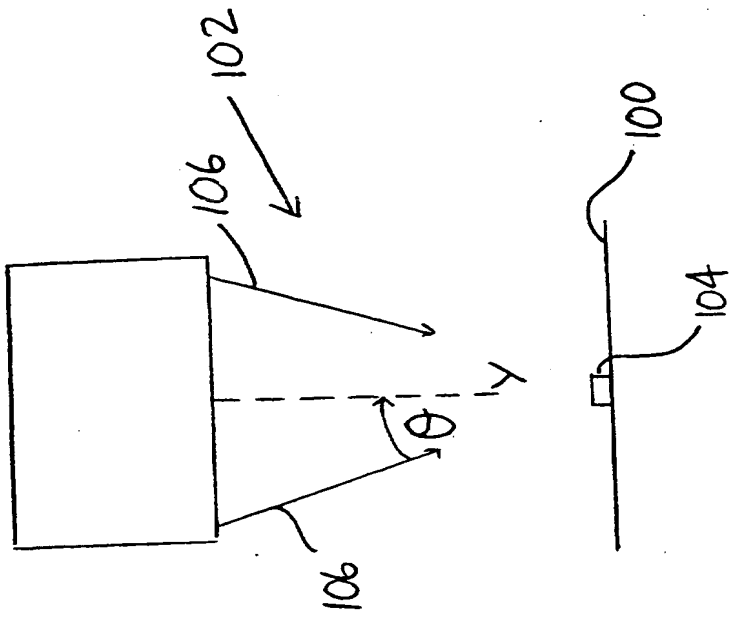

FIGURE 3

Diamond or Gemstone Marking

Background to the Invention

The present invention relates to a method and apparatus for marking a surface of a diamond or other gemstone. The mark may be any mark, but the invention is particularly but not exclusively directed to applying an information mark to the diamond. The diamond may be, for instance, an industrial diamond such as a wire-drawing die, though the invention is of particular interest in marking gemstone diamonds, and especially for applying a mark which is invisible to the naked eye or invisible to the eye using a x10 loupe (which is the loupe used by jewellers), when the mark can be applied to a polished facet of the gemstone without detracting from its clarity grade.

The marks can be used to uniquely identify the gemstone by a serial number or as a brand or quality mark, but it should not detract from the value or appearance of the stone, and should preferably not exhibit blackening.

There is a detailed description of the nature of the marks that can be applied in WO-97/03846, in which the marks are applied by irradiating a diamond gemstone with ultraviolet laser radiation using a projection mask.

It is generally desirable to produce marks of improved resolution and visibility when viewed using appropriate magnification and illumination conditions, the marks being such that they do not detract from the value and appearance of the diamond or other gemstone.

The Invention

In accordance with a first aspect of the present invention, there is provided a method of forming a mark on the surface of a diamond or gemstone, the mark consisting of one or more alphanumeric characters or the like, comprising the step of forming a plurality of grooves on the surface of the diamond or gemstone, the mark being such that it cannot be read by the naked eye, the grooves producing a visible diffraction effect under certain predetermined lighting conditions, such that the mark can be read under certain predetermined magnification conditions.

Also in accordance with the first aspect of the present invention, there is provided an apparatus for performing the above-mentioned method. The present invention further extends to a diamond or gemstone which has been marked by the above-mentioned method.

In accordance with the second aspect of the present invention, there is provided a method of forming a mark on the surface of a diamond or gemstone, comprising the step of forming a plurality of grooves on the surface of the diamond, said grooves producing a visible diffraction effect under certain predetermined illumination conditions without detrimentally affecting the clarity grade of the diamond or gemstone.

Also in accordance with the second aspect of the present invention, there is provided an apparatus for performing the above-mentioned method. The present invention further extends to a diamond or gemstone which has been marked by the above-mentioned method.

The greater the depth of the grooves, the more visible the mark will be when viewed. The grooves should be of a suitable depth so that the mark is highly visible under appropriate viewing conditions, but not so deep that the clarity grade of the diamond or other gemstone is detrimentally affected. In one preferred embodiment, each groove is

not less than about 10 nm deep and/or not more than about 50 nm deep with no evidence of blackening. A specific example would be around 30 nm.

The grooves may be in the form of parallel lines, or even a plurality of intersecting grooves forming cross-hatched pattern, depending on the effect desired.

Although the marking can be carried out using any suitable means, e.g. etching with an excimer laser or plasma etching, marking is preferably carried out using an ion beam, and most preferably by direct writing on the diamond surface with a focused ion beam. By limiting the dose, sputtering of carbon atoms can be avoided, sputtering causing direct material removal; this enables a mark to be applied with a controlled depth and good resolution. Typically Gallium ions are used, but a beam of other suitable ions may alternatively be used.

It is thought that each incident ion displaces a number of carbon atoms from their sites to create interstitials and vacancies in the diamond crystal. As the amount of damage (crystal lattice disorder) increases there is a tendency for the diamond sp^3 bonds to be replaced by the graphite like sp^2 bonds. These bonds can be attacked by a chemical etch to remove the disordered layer. By limiting the dose, and providing there is sufficient dose, the incident ions cause disordering that converts the diamond to a graphite-like or other non-diamond structure that can be cleaned using, for example, a powerful oxidizing agent, such as molten potassium nitrate, at a temperature of approximately 380-550 Centigrade for a period of between a few minutes and several hours.

The use of potassium nitrate has been found to be more effective in removing disordered diamond than other known processes, thus allowing a mark of a given depth to be produced with a relatively low dose of ions.

Other suitable oxidising agents may be molten compounds such as alkali metal salts; compounds in the form XnYm where the group X may be Li^+, Na^+, K^+, Rb^+, Cs^+ or

other cation, and the group Y may be OH^-, NO_3^-, O_2^{2-}, O^{2-}, CO_3^{2-} or other anion, the integers n and m being used to maintain charge balance. Mixtures of such compounds may be used. Air or other oxygen-containing gases may also be present.

As an alternative, the disordered layer of the diamond can be removed using an acid or potassium nitrate dissolved in acid. However, the use of, for example, molten potassium nitrate eliminates acid fumes. Furthermore, the need to dispose of spent acid is eliminated, thereby offering safety, environmental and economic benefits.

It is required to minimise the depth of disordering inflicted by the ion beam on the surface of the diamond. The depth of disordering is determined by the range of ions. For 50 keV Gallium, this range is about 30 nm. The minimum dose may be around 10^{13} / cm^2 and is preferably about $10^{14}/cm^2$ to $10^{15}/cm^2$, but good marks can be applied with a fairly modest dose, the preferred maximum dose being about $10^{16}/cm^2$ or even up to about $10^{17}/cm^2$. However, the dose depends upon the ions being used and their energy (as measured in keV). The ion beam dose is a total number of incident ions per unit area at the sample surface, during the marking. The beam current may be about 0.5 nA, and the beam energy not less than about 10 keV or about 30 keV and/or not greater than about 100 keV or about 50 keV.

It has been found that if depth of mark is plotted against ion beam dose for a series of different beam energies, there is an increase of depth of mark with increasing beam energy. Characteristics of the mark may be optimised by selecting from the dose/energy combinations which will result in the desired depth of mark.

The region to be marked and/or the surrounding area may be coated with an electrically conducting layer, for instance gold, prior to forming the mark, so that an electrical connection can be provided before marking with an ion beam, to prevent charging. The thickness of the gold, or other coating alters the variation of depth of the mark with beam energy and may thus be chosen to optimise the mark produced. However, it is preferred to irradiate the region to be marked with a low energy source of electrons

(e.g. around 1-100eV) from, for example, an electron flood gun, during the marking process to prevent charging.

If a focused ion beam is used to form the plurality of grooves, the accuracy of the method is such that no masking is required: the ion beam is applied directly to the surface of the diamond at the positions where the grooves are required to be formed. However, if other, less accurate methods of forming the grooves are to be used, then it may be necessary to mask the areas between the grooved areas to avoid marking them.

In accordance with the third aspect of the present invention, there is provided an apparatus for viewing a mark formed on a diamond using the method of the first or second aspect of the present invention, the apparatus comprising illumination means for illuminating the mark with directional light at an angle which corresponds to the diffraction angle of light of a predetermined wavelength or band of wavelengths, viewing means for viewing said mark, and magnification means for magnifying the viewed image of said mark. The invention also extends to a method of viewing the mark corresponding to the apparatus defined above.

The mark is preferably viewed against a dark background, ie. it is preferred that the illuminating light is substantially prevented from reflecting through the stone and appearing directly behind or close to the mark. It will be apparent to a person skilled in the art that, in order to achieve this, the angle and direction from which the illuminating light is supplied (and hence the orientation and spacing of the lines) must be chosen so as to ensure that no light can follow the undesired path.

The typical range of magnification required to view the mark is x10 to x50.

The distance between the plurality of grooves and the angle of the directional light determines the colour which the mark will appear when viewed. In general, for a diffraction grating:

$$d.\sin\theta = \pm n.\lambda$$

where d is the distance between each groove, θ is the angle of the incident light, λ is the wavelength of the diffracted light and n is an integer. Preferably n=1.

Thus, when a mark has been formed on a diamond, d and n are fixed, and the wavelength of diffracted light, i.e. the colour which the mark will appear when viewed, can be varied by varying the angle of incident light. Thus, if it is desired that the mark appears blue when viewed, then the angle of the incident light, i.e. θ, is set so that λ is around 450nm, using the above equation. Similarly, if the mark is to appear red, then θ is set so that λ is around 620nm.

In one embodiment of the apparatus according to the third aspect of the present invention, the illuminating means may comprise a light source and an opaque screen located in the incident light path, the screen having two apertures formed therein, the apertures being formed on either side of a generally central position such that two angular directional light sources are produced. The distance between the apertures determines the angle of the incident light sources. However, it is envisaged that the directional light source(s) may be provided by any convenient means, e.g. two separate light sources. A conventional microscope may include illumination means comprising a circular ring-shaped source comprising optical fibres illuminated by a remote tungsten light bulb. The illumination means of the present invention can be obtained by masking off all but two diametrically opposite sections of the illuminator.

Preferred Embodiment

Embodiments of the present invention will now be described by way of examples only and with reference to the accompanying drawings, in which:

Figure 1 is a magnified schematic diagram of the type of mark which is formed using the method and apparatus of the first and second aspects of the present invention;

Figure 2 is a further magnified cross-sectional view along line A - A of Figure 1; and

Figure 3 is a schematic view of an embodiment of apparatus according to the third aspect of the present invention.

Referring to Figure 1 of the drawings, a mark in the form of an alphanumeric character may be formed by means of a plurality of equally spaced, parallel elongate grooves 10 each separated by a distance d. Each groove 10 may have a generally square or rectangular cross-section, as shown in Figure 2. Alternatively, a sinusoidal profile may be preferred to reduce unwanted higher order diffraction.

Example

A specific method of forming each groove will now be described.

A diamond gemstone is mounted in a suitable holder and placed in a vacuum chamber equipped with a focused ion beam source such as supplied by FEI or Micrion. During exposure, the region to be marked may be irradiated using an electron flood gun supplied by Micrion, providing a low energy, e.g. 1-100 eV, source of electrons, to prevent the diamond from becoming charged.

Using a focused ion beam with a raster scan or similar to scan the beam with, for instance, electrostatic deflection (as an alternative, the diamond may be moved but this is less practical), and optionally any suitable software for controlling the ion beam, a series of closely spaced parallel lines are 'written' on the diamond facet.

The sample is removed from the vacuum chamber, placed in a stainless steel crucible, and covered with a powerful oxidising agent, such as molten potassium nitrate, for a

period of around one to two hours. The sample is subsequently cooled and removed from the potassium nitrate before being cleaned using water and ethanol, thereby removing the portions of the diamond surface which have been disordered by the ion beam, and leaving a series of closely spaced grooves each around 30 - 35 nm deep, with no evidence of blackening.

Upon examination before cleaning, the exposed region is identifiable by its graphite-like appearance when examined, for example, in a reflected light microscope. Such a mark would not be acceptable to a diamond grader, in that it would substantially reduce the clarity grade of the diamond. However, after cleaning using the powerful oxidising agent, the mark is not easily visible in a microscope, with no contrast between the mark and surrounding areas. The mark only becomes visible when illuminated by preferably two directional light sources at an angle which corresponds to the angle of diffracted light of a particular wavelength, for example blue light, at which time the mark appears blue. Such a mark is acceptable to a diamond grader in that it does not detrimentally affect the clarity grade of the diamond.

The closely spaced grooves are preferably formed within an 'invisible outline' of an alphanumeric character or the like, as shown in Figure 1 of the drawings.

Referring now to Figure 3 of the drawings, a method and apparatus for viewing the mark produced by the process described above will now be described by way of example only.

The marked diamond 104 is placed on the viewing surface 100 of a conventional microscope 102. The diamond 104 is illuminated by two directional light sources 106 having an angle θ relative to the vertical axis Y. As described above, θ is chosen so that the mark appears to be, for example, blue or red, as desired. Thus, if the mark is to appear blue, and d is approximately 1200nm, then θ is chosen to satisfy:

$$d.\sin\theta = \text{(approximately) } 450\text{nm}$$

where n=1 and 450nm is the approximate wavelength of blue light, which is the wavelength of the diffracted light at X in Figure 3. In this case, $\theta = 22°$.

The directional light sources may be provided by a generally ring-shaped illuminator, all but two diametrically opposite portions thereof being masked off. However, any suitable light source may be used to produce the same effect.

The present invention has been described above purely by way of example, and modifications can be made within the spirit of the invention, which extends to the equivalents of the features described. The invention also consists in any individual features described or implicit herein or shown or implicit in the drawings or any combination of any such features or any generalisation of any such features or combination.

CLAIMS:

1. A method of forming a mark on a surface of a diamond or gemstone, the mark consisting of one or more alphanumeric characters or the like, comprising the step of forming a plurality of grooves on said surface of the diamond or gemstone, the mark being such that it cannot be read by the naked eye, the grooves producing a visible diffraction effect under certain predetermined lighting conditions such that the mark can be read under certain predetermined magnification conditions.

2. A method according to claim 1, wherein said grooves are formed by means of a focused ion beam.

3. A method according to claim 2, wherein areas of said surface of the diamond or gemstone in which the crystal lattice thereof is disordered by said focused ion beam are removed by means of a highly oxidising agent.

4. A method according to claim 3, wherein said highly oxidising agent is molten potassium nitrate.

5. A method according to claim 3, wherein said highly oxidising agent is a compound in the form XnYm, where the group X is Li^+, Na^+, K^-, Rb^+, Cs^+ or other cation, or mixtures thereof, and the group Y is OH^-, NO_3^-, O_2^{2-}, O^{2-}, CO_3^{2-} or other anion, or mixtures thereof, the integers n and m being used to maintain charge balance.

6. A method according to any preceding claim, wherein said grooves are formed as substantially equally spaced apart, parallel elongate grooves.

7. A method according to claim 6, wherein a plurality of intersecting sets of grooves are formed so as to produce a cross-hatched effect.

8. A method according to any one of claims 2 to 7, wherein said focused ion beam has a beam energy of 50 keV or less.

9. A method according to any one of claims 2 to 8, wherein said focused ion beam has a beam current of about 0.5 nA.

10. A method according to any one of claims 2 to 9, wherein the dosage of ions provided by said focused ion beam is approximately $10^{13}/cm^2$ to $10^{17}/cm^2$.

11. A method according to claim 10, wherein said dosage is approximately $10^{14}/cm^2$ to $10^{16}/cm^2$.

12. A method according to any one of claims 2-11, wherein the region of said surface of a diamond or gemstone to be marked is irradiated with a low energy source of electrons, in order to eliminate charging.

13. A method according to any one of claims 1 to 12, wherein said diamond is a gemstone diamond.

14. Apparatus for forming a mark on a surface of a diamond or gemstone, the mark consisting of one or more alphanumeric characters or the like, comprising means for forming a plurality of grooves on said surface of the diamond or other gemstone, the mark being such that it cannot be read by the naked eye, the grooves producing a visible diffraction effect under certain predetermined lighting conditions such that it can be read under certain predetermined magnification conditions.

15. Apparatus according to claim 14, wherein said means for forming said plurality of grooves comprises means for irradiating portions of said diamond or gemstone so as to cause the crystal lattice thereof to become disordered and produce a disordered layer, and means for removing said disordered layer.

16. Apparatus according to claim 15, wherein said means for irradiating portions of said diamond comprises a focused ion beam.

17. Apparatus according to claim 15 or claim 16, wherein said disordered layer is removed by means of a highly oxidising agent.

18. Apparatus according to claim 17, wherein said highly oxidising agent is molten potassium nitrate.

19. Apparatus according to claim 17, wherein said highly oxidising agent is a compound in the form XnYm, where the group X is Li^+, Na^+, K^+, Rb^+, Cs^+ or other cation, or mixtures thereof, and the group Y is OH^-, NO_3^-, O_2^{2-}, O^{2-}, CO_3^{2-} or other anion, or mixtures thereof, the integers n and m being used to maintain charge balance.

20. Apparatus according to any one of claims 15 to 19, wherein said focused ion beam has a beam energy of 50 keV or less.

21. Apparatus according to any one of claims 15 to 20, wherein said focused ion beam has a beam current of about 0.5 nA.

22. Apparatus according to any one of claims 15 to 21, wherein the dosage of ions provided by said focused ion beam is approximately $10^{13}/cm^2$ to $10^{17}/cm^2$.

23. Apparatus according to claim 22, wherein said dosage is approximately $10^{14}/cm^2$ to $10^{16}/cm^2$.

24. Apparatus according to any one of claims 14 to 23, further comprising means for irradiating the region of said surface of a diamond to be marked with a low energy source of electrons, in order to eliminate charging.

25. Apparatus according to claim 24, wherein said means for irradiating is an electron flood gun.

26. A method or apparatus according to any preceding claim, wherein said mark does not detrimentally affect the clarity grade of the diamond.

27. A diamond or gemstone having a mark formed thereon by means of the method according to any one of claims 1 to 13.

28. A method of forming a mark on the surface of a diamond or gemstone, comprising the step of forming a plurality of grooves on the surface of the diamond or gemstone, said grooves producing a visible diffraction effect under certain predetermined illumination conditions, without detrimentally affecting the clarity grade of the diamond or gemstone.

29. A method according to claim 28, wherein said mark is in the form of one or more alphanumeric characters or the like.

30. A method according to claim 28 or claim 29 wherein, said grooves are formed by means of a focused ion beam.

31. A method according to claim 29 or claim 30, wherein areas of said surface of said diamond or gemstone in which the crystal lattice thereof is disordered by said focused ion beam are removed by a highly oxidising agent.

32. A method according to claim 31, wherein said highly oxidising agent is molten potassium nitrate.

33. A method according to claim 31, wherein said highly oxidisable agent is a compound in the form XnYm, where the group X is Li^+, Na^+, K^+, Rb^+, Cs^+ or other

cation, or mixtures thereof, and the group Y is OH^-, NO_3^-, O_2^{2-}, O^{2-}, CO_3^{2-} or other anion, or mixtures thereof, the integers n and m being used to maintain charge balance.

34. A method according to any one of claims 28 to 33, wherein said grooves are formed as substantially equally spaced apart, parallel elongate grooves.

35. A method according to claim 34, wherein two or more intersecting sets of grooves are formed so as to produce a cross-hatched effect.

36. A method according to any one of claims 29 to 35, wherein said focused ion beam has a beam energy of 50 keV or less.

37. A method according to any one of claims 29 to 36, wherein said focused ion beam has a beam current of about 0.5 nA.

38. A method according to any one of claims 29 to 37, wherein the dosage of ions provided by said focused ion beam is approximately $10^{13}/cm^2$ to $10^{17}/cm^2$.

39. A method according to claim 38, wherein said dosage is approximately $10^{14}/cm^2$ to $10^{16}/cm^2$.

40. A method according to any one of claims 28 to 39, wherein the region of said surface of a diamond or gemstone to be marked is irradiated with a low energy source of electrons, in order to eliminate charging.

41. A diamond having a mark formed thereon by means of the method according to claims 28 to 40.

42. Apparatus for forming a mark on the surface of a diamond or gemstone, comprising means for forming a plurality of grooves on the surface of the diamond, said grooves producing a visible diffraction effect under certain predetermined

illumination conditions, without detrimentally affecting the clarity grade of the diamond.

43. Apparatus according to claim 42, wherein said means for forming said plurality of grooves comprises means for irradiating portions of said diamond or gemstone so as to cause the crystal lattice thereof to become disordered and produce a disordered layer, and means for removing said disordered layer.

44. Apparatus according to claim 43, wherein said means for irradiating portions of said diamond or gemstone comprises a focused ion beam.

45. Apparatus according to claim 43 or claim 44, wherein said disordered layer is removed by means of a highly oxidising agent.

46. Apparatus according to claim 45, wherein said highly oxidising agent is molten potassium nitrate.

47. Apparatus according to claim 45, wherein said highly oxidising agent is a compound in the form XnYm, where the group X is Li^+, Na^+, K^+, Rb^+, Cs^+ or other cation, or mixtures thereof, and the group Y is OH^-, NO_3^-, O_2^{2-}, O^{2-}, CO_3^{2-} or other anion, or mixtures thereof, the integers n and m being used to maintain charge balance.

48. Apparatus according to any one of claims 44 to 47, wherein said focused ion beam has a beam energy of 50 keV or less.

49. Apparatus according to any one of claims 44 to 48, wherein said focused ion beam has a beam current of about 0.5 nA.

50. Apparatus according to any one of claims 44 to 49, wherein the dosage of ions provided by said focused ion beam is approximately $10^{13}/cm^2$ to $10^{17}/cm^2$.

51. Apparatus according to claim 50, wherein said dosage is approximately $10^{14}/cm^2$ to $10^{16}/cm^2$.

52. Apparatus according to any one of claims 42 to 51, further comprising means for irradiating the region of said surface of a diamond or gemstone to be marked with a low energy source of electrons, in order to eliminate charging.

53. Apparatus according to claim 52, wherein said means for irradiating is an electron flood gun.

54. Apparatus for viewing a mark on the surface of a diamond or gemstone, said mark consisting of a plurality of grooves which produce a highly diffractive effect when magnified and illuminated, the apparatus comprising illumination means for illuminating the mark with directional light at an angle which corresponds to the diffraction angle of light of a predetermined wavelength or band of wavelengths, viewing means for viewing said mark, and magnification means for magnifying the viewed image of said mark.

55. Apparatus according to claim 54, wherein the angle and direction from which the illuminating light is supplied is such that the mark can be viewed against a relatively dark background.

56. A method of viewing a mark on the surface of a diamond or gemstone said mark consisting of a plurality of grooves which produce a diffractive effect when illuminated, the method comprising the steps of illuminating the mark with directional light at an angle which corresponds to the diffraction angle of light of a predetermined wavelength or band of wavelengths, viewing said mark, and magnifying the viewed image of said mark.

57. A method according to claim 56, comprising the step of choosing the angle and direction from which the illuminating light is supplied so as to view the mark against a relatively dark background.

58. A method of forming a mark on a surface of a diamond or gemstone substantially as herein described with reference to the accompanying drawings.

59. Apparatus for forming a mark on a surface of a diamond or gemstone substantially as herein described with reference to the accompanying drawings.

60. Apparatus for viewing a mark formed on a surface of a diamond or gemstone, substantially as herein described with reference to the accompanying drawings.

61. A method of viewing a mark formed on the surface of a diamond or gemstone, substantially as herein described with reference to the accompanying drawings.

Application No: GB 9828393.0 **Examiner:** Graham Russell
Claims searched: 1-61 **Date of search:** 12 March 1999

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.Q): B6J (JMY); G2J

Int Cl (Ed.6): B23K 26/00; B44B 7/00

Other: Online: EPODOC, PAJ, WPI

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
X	GB 718651	(ICI) see Fig 1 and page 2 lines 10-46	54
X	EP 648445 A1	(YAMATO) see page 5 lines 19-35 & Fig 1(a)	28,42
X	WO 94/20837 A1	(GERSAN) see Fig 1 and page 15 line 4 - page 16 line 15	54
X	US 4906083	(SATTLER) see Fig 1 and column 10 lines 61-62	54

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.